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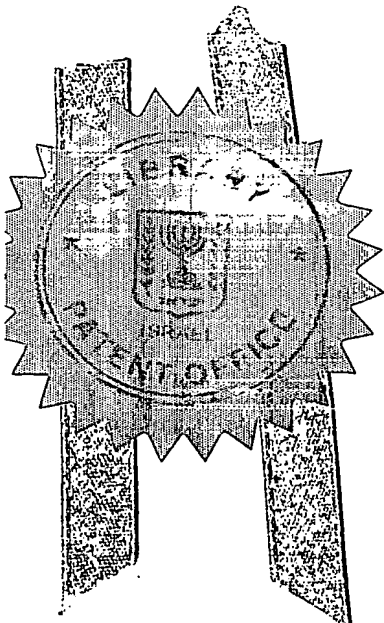
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שיטה למיצוי ערכי בריליום באמצעות חומצה הידרופלואורית

(בעברית)
(Hebrew)

METHOD OF LEACHING BERYLLIUM VALUES USING
HYDROFLUORIC ACID

(באנגלית)
(English)

hereby apply for a patent to be granted to me in respect thereof

מבקש בזאת כי ינתן לי עליה פטנט

• בקשת חלוקה - Application of Division		• בקשת פטנט מוסף - Application for Patent Addition		• דרישת דין קדימה Priority Claim		
מבקשת פטנט from Application	מס' No.	לבקשה/לפטנט to Patent/.Appl	מס' No.	מספר/סימן Number/Mark	תאריך Date	מדינת האיגוד Convention Country
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המען למסירת מכתבים בישראל Address for service in Israel .DR. MARK FRIEDMAN LTD BEIT SAMUELOFF 7HAOMANIM STREET 67897TEL AVIV						
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שיטה למיצוי ערכי בריליום באמצעות חומצה הידרופלואורית

METHOD OF LEACHING BERYLLIUM VALUES USING HYDROFLUORIC ACID

FIELD OF THE INVENTION

The present invention relates to a method of leaching beryllium values from beryllium-containing ores using hydrofluoric acid in an aqueous medium.

5

BACKGROUND OF THE INVENTION

Beryllium (Be) is one of the lightest of all metals and has one of the highest melting points of any light metal. Beryllium metal and its alloys are used principally in aerospace and defense applications because of its stiffness, low specific gravity, and dimensional stability over a wide temperature range. Beryllium metal is stiffer than 1010 steel, and is also highly conductive. Beryllium and alloys thereof are often the preferred materials for electronic equipment. Beryllium-copper alloys are used in a variety of applications because of their electrical and thermal conductivity, high strength and hardness, good corrosion resistance and fatigue resistance, and non-magnetic properties. Beryllium oxide is an excellent heat conductor, with high strength and hardness, and acts as an electrical insulator in some applications.

Commercially, beryllium is extracted from mineral ores, primarily from beryl, bertrandite and phenakite. The composition of beryl is $3\text{BeO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$. Commercial Beryl contains about 12% BeO, which is close to the theoretical composition of beryl (14% BeO). The composition of bertrandite is $4\text{BeO} \cdot 2\text{SiO}_2 \cdot \text{H}_2\text{O}$, which, according to the theoretical chemical composition, contains 42% BeO. Commercially-available bertrandite contains only about 0.6%-0.9% BeO. Whereas the commercially-available beryl is

almost a pure mineral, the commercially-available bertrandite contains only 1.0-1.5% of the pure bertrandite, admixed with additional minerals such as aluminum and iron oxides, calcium and alkali oxides or silicates and quartz.

The domestic (U.S.A.) producer price for beryllium contained in beryllium-copper master alloy was \$160 per pound in 1998, a price level that was relatively steady since 1990. Prices for other forms of beryllium have also been stable for nearly a decade. Beryllium and beryllium alloys are very often the most expensive materials available for a given application. Hence, the use of metal beryllium and compounds thereof is often precluded in all but the most exotic applications.

One major factor behind the cost of beryllium products is high production costs. Ullmann's Encyclopedia of Industrial Chemistry (Vol. 4a, Wiley-VCH Verlag GmbH & Co., pp. 15-26 (1985), which is incorporated by reference for all purposes, as if fully set forth herein, surveys the known methods for treating and dissolving beryllium ores, and for processing them into various desired beryllium compounds.

In the production of beryllium from bertrandite, boiling sulfuric acid is used to dissolve the bertrandite, along with various oxide impurities (e.g., aluminum, iron) typically present in the commercial ore. The solution is filtered to remove any insoluble silica from the solution.

One important drawback of bertrandite as a feed material is that commercially-available bertrandite contains less than 1% BeO, i.e., only 5-8% of the BeO content of commercially-available beryl.

However, the processing and dissolution of the beryllium-rich beryl is considerably more complicated, expensive, and involves additional, weighty, health and safety issues. Ullmann's Encyclopedia reports that beryl is only slightly soluble in sulfuric acid even under such extreme conditions as heating
5 for several hours in an autoclave at 400°C, hence the beryl must be subjected to either an alkali or a heat treatment.

In the alkali treatment, finely ground beryl is heated until fusion or sintered below the melting point with a sufficient quantity of alkaline flux. Suitable alkali compounds include hydroxides or carbonates of sodium,
10 potassium and calcium; mixtures of these carbonates; calcium oxide; borax; lead chloride; and sodium sulfate and charcoal. The ratio of flux to beryl depends on the operating conditions, especially the temperature: the higher the temperature the less flux. For alkali treatments involving molten material, gas-heated or oil-heated rotary furnaces or blast furnaces are suitable. Rotary kilns,
15 muffle furnaces, or tunnel kilns are used for sintering.

In the heat treatment, the beryl is melted, without additives, at a temperature exceeding 1650°C, and then quenched in water. After this treatment, about 50-60% of the beryl has an enhanced solubility in sulfuric acid. The rest of the beryllium oxide forms a solid solution with silicon
20 dioxide that is not attacked by sulfuric acid. At 900°C, this solid solution separates into beryllium oxide and silicon dioxide components, hence, a second heat treatment at this high temperature produces a free beryllium oxide that is soluble in sulfuric acid.

After cooling and grinding to 200 mesh, the obtained powder is heated to 250°C to 300°C in concentrated sulfuric acid, reacting so as to convert the beryllium, aluminum, and any iron, to soluble sulfates. The silica fraction largely remains in the dehydrated, water insoluble form. This two-stage heat treatment renders soluble a total of 90-95% of the beryl.

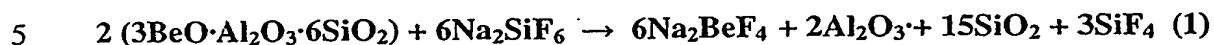
The heat-treated beryl is extracted with hot concentrated sulfuric acid, whereas alkali-treated beryl is extracted with cold sulfuric acid. While in the alkali process, there are relatively low heating costs, the total consumption of acid is considerably greater because of the alkali added.

After dilution of the liquor with water, the insoluble silicon dioxide is separated by filtration. At this point in the process, the filtrate contains not only beryllium and aluminum sulfate but also considerable quantities of iron sulfate and smaller amounts of other impurities, all of which must be removed before the precipitation of beryllium hydroxide. Many separation processes are known, including alum separation, ammonium carbonate separation, and chelate separation.

It is manifest from the above that numerous time-consuming and expensive steps are required to dissolve the beryl ore so as to obtain dissolved beryllium. Moreover, many additional steps are subsequently required to obtain high purity beryllium and beryllium oxide from the impure beryllium solution.

Although other processes for the decomposition of beryl are known, such as the fluoride process, these have generally proven to be even more

complicated and expensive than the sulfuric acid process. In the conventional fluoride process, beryl is melted or sintered with sodium hexafluorosilicate at approximately 700°C, according to the stoichiometry of the following equation:



Aluminum oxide, silicon dioxide, and other impurities remain largely intact. Ullmann's Encyclopedia further discloses (based on Derwin, et al., Beryllium, Butterworths, London, 1960) that the subsequent leaching step is extremely delicate:

10 The reaction product must be leached with water at room
 temperature because silicon dioxide is attacked by
 fluoride solutions at elevated temperatures. On the other
 hand, the leaching must be performed as rapidly as
15 possible because otherwise the beryllium salt that is
 already dissolved will precipitate and in this form it is
 sparingly soluble, unlike the form which is present in the
 original fusion product.

In addition, the melting process at elevated temperatures (~700°C) is a clear
disadvantage, although the sulfate process described hereinabove requires even
20 higher temperatures.

Other suitable extracting agents mentioned by Ullmann's Encyclopedia
(Vol. A4, p.18) are hydrogen fluoride gas at 630°C, molten fluorides, other
fluorosilicates, or silicon tetrafluoride. Rotary kilns are used for reaction with
the gases (hydrogen fluoride, silicon tetrafluoride); the solid mixtures (molten

fluorides, other fluorosilicates) are briquetted and sintered in muffle furnaces or tunnel kilns.

U.S. Patent No. 3,375,060 to Olson, et al. discloses a method of solubilizing beryllium values in a mineral by reacting the mineral with fluorite
5 (CaF₂) and sulfuric acid. The reaction takes place at a temperature of 200°C - 300°C and atmospheric pressure. The products of the reaction are gaseous hydrogen fluoride and a salt cake containing water soluble beryllium sulfate along with calcium sulfate, aluminum sulfate, iron sulfate and various impurities. Separation of beryllium values from the solid calcium sulfate is
10 subsequently performed by leaching with water.

At atmospheric pressure, operation at elevated temperatures of 200°C - 300°C is possible only due to the boiling-point elevation provided by a great excess of concentrated sulfuric acid. Indeed, U.S. Patent No. 3,375,060 stipulates that the concentration of the sulfuric acid feed material must be at
15 least 60% by weight, and preferably at least 96% by weight. Moreover, the feed ratio of sulfuric acid to solid feed (i.e., the weight of H₂SO₄ per unit weight of fluorite and beryllium-containing mineral) must be at least 1 to 1, and preferably about 1.4 to 1.

Various disadvantages are manifest in this process, including high
20 temperature and severe reaction conditions, downstream processing of the excess sulfuric acid, ecological issues pertaining to the impurity-containing CaSO₄ by-product cake, the calcination process at 500°C - 600°C to render iron and aluminum less soluble, and the concentration of the very dilute beryllium-

containing solution. In addition, the hydrogen fluoride co-product limits the applicability of the process.

U.S. Patent No. 3,375,060 to Zimmermann teaches a method of decomposing beryl and beryl-containing minerals by reacting powdered beryl
5 with gaseous hydrogen fluoride at 100°C - 900°C. Preferably, the reaction is performed at 500°C - 600°C.

The products of the reaction are SiF_4 vapor (which is condensed with water to produce SiO_2) and a cake containing water-soluble beryllium fluoride along with aluminum fluoride, iron fluoride and various impurities. Separation
10 of beryllium values from the cake is subsequently performed by leaching with hot water.

The main disadvantages in the above-described process relate to the use of hydrogen fluoride in the vapor phase. Operation at elevated temperatures of 100°C - 900°C, and preferably, at 500°C - 600°C, requires extremely high
15 pressures, tremendous superheating, or both. In the case of superheating, an extremely large reaction volume is required. Operation at elevated pressures requires special equipment. In particular, operation above about 200°C requires exotic materials of construction that render the process impractical.

Moreover, these extreme operating conditions raise various safety and
20 ecology issues related to the processing of beryllium compounds. Although solid beryllium poses no health hazard, inhaling beryllium particles or fumes can trigger acute or chronic lung disease. Chronic beryllium disease, or berylliosis, is an allergic reaction to beryllium resulting in inflammation and

scarring of the lungs and inhibiting oxygenation of the bloodstream. Symptoms include shortness of breath, dry cough, chest pain, fatigue, weight loss and loss of appetite.

Exposure to beryllium for an eight-hour work period should not exceed 2
5 micrograms of beryllium/m³. A brief exposure should not exceed 25
micrograms/m³.

There is therefore a recognized need for, and it would be highly
advantageous to have, a simple and economical process for producing
dissolved beryllium that overcomes the numerous and manifest deficiencies of
10 the processes known in the art. It would be of further advantage to have a
process that is safer and more ecologically friendly with respect to these known
processes.

15

BRIEF DESCRIPTION OF THE DRAWINGS

The invention is herein described, by way of example only, with
reference to the accompanying drawings. With specific reference now to the
20 drawings in detail, it is stressed that the particulars shown are by way of
example and for purposes of illustrative discussion of the preferred
embodiments of the present invention only, and are presented in the cause of
providing what is believed to be the most useful and readily understood
description of the principles and conceptual aspects of the invention. In this
25 regard, no attempt is made to show structural details of the invention in more

detail than is necessary for a fundamental understanding of the invention, the description taken with the drawings making apparent to those skilled in the art how the several forms of the invention may be embodied in practice.

In the drawings:

5 Figure 1 is a block diagram of the inventive method for processing a beryllium feed source with hydrofluoric acid in an aqueous medium.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

10

The present invention is a method of treating a beryllium feed material, such as beryl, by reacting the beryllium feed material with hydrofluoric acid in an aqueous medium.

15 The principles and operation of the method according to the present invention may be better understood with reference to the drawings and the accompanying description.

20 Before explaining at least one embodiment of the invention in detail, it is to be understood that the invention is not limited in its application to the details of construction and the arrangement of the components set forth in the following description or illustrated in the drawing. The invention is capable of other embodiments or of being practiced or carried out in various ways. Also, it is to be understood that the phraseology and terminology employed herein is for the purpose of description and should not be regarded as limiting.

25 Reference is now made to Figure 1, in which a method for the reactive dissolution of beryllium from a beryllium feed source 6 is schematically

presented. Beryllium feed source 6 is admixed with hydrofluoric acid 8 in reaction stage 10. The beryllium feed source 6 largely or completely dissolves, leaving the highly soluble beryllium values (largely) in the liquid phase. Most of the other species are also dissolved in the liquid phase. If necessary, the reaction product mixture 18 produced in reaction stage 10 is subsequently subjected to a solid/liquid separation 20, to produce a residue 14 and a beryllium-rich solution 16. Residue 14 may be processed for further retrieval of beryllium values, or may be discarded.

It must be emphasized that the production process of the present invention is appropriate for both batch and continuous modes of operation.

Typically, beryllium feed source 6 is a commercially-available ore containing beryl ($3\text{BeO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$), bertrandite ($4\text{BeO} \cdot 2\text{SiO}_2 \cdot \text{H}_2\text{O}$), and/or phenakite (Be_2SiO_4). Often, such commercially-available ores include additional minerals containing aluminum and iron oxides, calcium and alkali oxides or silicates and quartz. In reaction stage 10, various aluminum, silica, and iron-based minerals from beryllium feed source 6 tend to dissolve.

Of the various beryllium-containing ores, beryl is known to be the most difficult to dissolve. Henceforth, beryllium feed source 6 will refer specifically to beryl.

While sulfuric acid 12 may be added to reaction stage 10 to promote the dissolution of beryllium feed source 6, the addition of sulfuric acid 12 has been found to be unnecessary, as the dissolution proceeds essentially to completion at atmospheric or near-atmospheric pressures and at a temperature of about

100°C. Preferably, the temperature in the reaction stage is at least 80°C, and more preferably, above about 100°C.

When sulfuric acid 12 is added to the beryl (beryllium feed source 6), the preferred temperature range for effecting the dissolution is 100°C - 250°C.

5 However, because of the aggressive reaction conditions, high sulfuric acid concentrations (and/or super-atmospheric pressures), and other costs and concerns pertaining to the operation at the higher end of this range, it appears to be preferable to perform the reaction below a temperature of about 200°C.

The hydrofluoric acid reacts with SiO₂ values to produce fluosilicic acid
10 (H₂SiF₆) and water according to the following stoichiometry:



or alternatively,



Reaction stage 10 can be operated such that vapor stream 13, which may
15 contain hydrofluoric acid, silicon tetrafluoride and water, is removed from reaction stage 10. Alternatively, vapor stream 13 can be refluxed back to the reaction mixture in reflux stream 15.

In another preferred embodiment, the dissolution of beryllium feed source 6, and more specifically, beryl, is performed in a sealed vessel,
20 autoclave, or the like (reaction stage 10), which is hermetically sealed from the environment (i.e., there is no vapor stream 13 being discharged from reaction stage 10). Preferably, substantially all of the reaction volume is filled with the reaction mixture. Substantially complete dissolution of the beryl can be

effected using hydrofluoric acid 8, added in excess. Under such conditions, no addition of sulfuric acid 12 is needed. With increasing temperature, and for a fixed residence time, the fraction of dissolved beryllium increases.

Upon completion of reaction stage 10, a mixture of (excess) hydrofluoric acid and fluosilicic acid, together with the products of dissolution, is typically obtained. This mixture can be separated, and all the acids recovered, according to various processes known in the art.

In another aspect of the present invention, however, a second beryllium source 26 is introduced to a second reaction stage 30 along with reaction product mixture 18 so as to dissolve beryllium values present in second beryllium source 26 as well as to allow additional beryllium values to be leached from any residue remaining from reaction stage 10. This is particularly advantageous in that at least a portion, and preferably substantially all of the excess acid from reaction stage 10 can be consumed, liberating additional beryllium values and perhaps more importantly, obviating the need for the above-mentioned hydrofluoric acid — fluosilicic acid separation.

Second beryllium source 26 preferably includes bertrandite, which has been found by the inventors to dissolve readily (substantially 100%) in the presence of these acids at moderate temperatures as low as 60°C - 110°C. Other beryllium feed sources that dissolve more readily than beryl may also be used in second beryllium source 26.

As used herein in the specification and in the claims section that follows, the term "beryllium feed source" refers to a raw material, or combination of

raw materials, containing beryllium. "Beryllium feed source" specifically includes, but is not limited to, materials containing beryl, bertrandite, and/or phenakite.

As used herein in the specification and in the claims section that follows,
5 the term "readily soluble beryllium feed source" refers to a beryllium feed source that includes, but is not limited to, bertrandite, and/or phenakite. The term "readily soluble beryllium feed source" is meant to specifically exclude beryl.

As used herein in the specification and in the claims section that follows,
10 the term "directly introduced to the reaction stage" and the like, used in conjunction with "beryllium feed source", refers to a beryllium feed source that is reacted with hydrofluoric acid, without first undergoing melting or other high-temperature treatments, and particularly, without undergoing high-temperature treatment at temperatures exceeding 600°C.

15 The particular operating conditions of the process of the present invention will understandably vary according to the composition of beryllium feed source 6, local process conditions, etc.

Advantages of the Inventive Process

- 20
- In the known sulfate process for the production of beryllium compounds from beryl, described hereinabove, the multiple-stage thermal treatment is both capital-intensive and energy-intensive. By sharp contrast, the dissolution of beryllium oxide by means of hydrofluoric acid requires no prior thermal treatment of the ore
25 whatsoever, and minimal heating for the dissolution stage

- 5 • The high toxicity of beryllium introduces various safety and ecology issues into the production process. The inhalation of beryllium particles or fumes can trigger acute or chronic lung disease of a serious nature. The inventive process obviates the need for the high-temperature, multiple-stage thermal treatment, significantly reducing thereby the health risk to plant personnel and/or reducing the costs associated with maintaining the requisite high standard of air quality for beryllium production facilities.
- 10 • In other known processes, a large excess of concentrated sulfuric acid is utilized for the dissolution process, and must be neutralized later in the process. In the present invention, sulfuric acid is not necessary, and certainly not in large quantities. The cost of the sulfuric acid and the neutralizing substance are saved, and the ecological benefits are manifest.
- 15 • The leaching of the beryllium oxides from the ores by sulfuric acid occurs at 250-300°C in conventional processes, whereas the inventive process is effective at relatively low temperatures (near 100°C).

20 Incorporated by reference for all purposes as if fully set forth herein are Israeli Patent Application Serial Nos. IL 140,646, IL 144,038, IL 148,376, and IL 152,802, as well as PCT Application No. PCT/IL01/01210, which draws priority from IL 148,376.

25

EXAMPLES

Reference is now made to the following examples, which together with the above description, illustrate the invention in a non-limiting fashion.

EXAMPLES 1-10

5 Samples of beryl were reacted with hydrofluoric acid (38% HF) in aqueous media for 1-6 hours at temperatures between 100°C and 110°C.

 The finely-ground beryl used in Example 1 contained: 12.18% BeO, 0.03% CaO, 0.86% Fe₂O₃, 0.73% Na₂O, 17.44% Al₂O₃, 0.22% CoO, 0.12% MgO, 0.39% PbO, 64.45% SiO₂, 0.03% TiO₂, 0.42% Cr₂O₃.

10 The finely-ground beryl used in Example 1 contained: 13.08% BeO, 17.25% Al₂O₃, 63.91% SiO₂, 0.91% Fe₂O₃, 0.19% MgO, 0.7% Na₂O, 1.4% ZrO, 0.35% PbO.

 In Examples 4-10, the evaporate was completely refluxed (see stream 15 of Figure 1) to the reaction mixture. The results are summarized in Table 1.

EXAMPLE 11: Dissolution of beryl in a sealed capsule

15 The beryl used in Example 11 contained 13.08% BeO, 17.25% Al₂O₃,
20 and 0.91% Fe₂O₃.

 To a Teflon® (polytetrafluoroethylene)-lined capsule having a reaction volume of 60 ml were added 12.2 grams of beryl and 58 ml of hydrofluoric acid (38% HF), such that the entire volume of the capsule was filled with the reaction mixture. The capsule was hermetically sealed and placed in an oven
25 that was heated to 125°C for 20 hours. The capsule was then cooled and

opened. The content of the capsule was filtered, the solid residue was washed, dried and weighed. The results were as follows:

dry residue weight: 3.01g

filtrate and washing liquor weight: 296 g

5 BeO concentration in the solution: 5.25g/l

amount of BeO in solution: $296 \times 5.25 / 1000 = 1.554 \text{ g}$

amount of BeO in original Beryl: $12.2 \times 13.08 / 100 = 1.596 \text{ g}$

BeO yield: $1.554 / 1.596 \times 100 = 97.3\%$

10 The results of Example 11 are summarized in Table 2.

Although the invention has been described in conjunction with specific embodiments thereof, it is evident that many alternatives, modifications and variations will be apparent to those skilled in the art. Accordingly, it is
15 intended to embrace all such alternatives, modifications and variations that fall within the spirit and broad scope of the appended claims. All publications, patents and patent applications mentioned in this specification are herein incorporated in their entirety by reference into the specification, to the same extent as if each individual publication, patent or patent application was
20 specifically and individually indicated to be incorporated herein by reference. In addition, citation or identification of any reference in this application shall not be construed as an admission that such reference is available as prior art to the present invention.

Table 1

	T (°C)	Duration (h)	% Excess HF ¹	Dissolved Solids (%)	Be Yield (%)
EXAMPLE 1	97	1.5	100	78	72.1
EXAMPLE 2	108	4	57	86	86
EXAMPLE 3	102	4	561	91.4	91.9
EXAMPLE 4	97	4	367	97.9	99.3
EXAMPLE 5	108	3	340	99.1	96.9
EXAMPLE 6	105	3	238	99.2	93.3
EXAMPLE 7	104	3	120	94.2	89.5
EXAMPLE 8	108	2	340	97.6	91.1
EXAMPLE 9	107	1	340	88.2	81.7
EXAMPLE 10	105	6	61	80.4	94.6

¹% Excess HF calculated on all ore components, including silica

5

TABLE 2

Example No.	Beryl quantity (grams)	Acid quantity (ml)	Excess ¹ (%)	Temperature (°C)	Duration (hours)	Residue (grams)	Dissolution of Be (%)
11	12.2	58.5	61	125	20	3.01	97.3

¹% Excess HF calculated on all ore components, including silica

10

WHAT IS CLAIMED IS:

1. A production process for dissolving beryllium in a beryllium feed source by means of hydrofluoric acid in an aqueous medium, the process comprising the steps of:

- (a) providing the beryllium feed source;
- (b) reacting the beryllium feed source with the hydrofluoric acid in a reaction stage to produce dissolved beryllium values in the aqueous medium, and
- (c) processing said dissolved beryllium values to produce a refined beryllium-containing product.

2. The process according to claim 1, wherein said reaction stage yields a solid residue along with the aqueous medium, the process further comprising the step of:

- (d) separating at least a portion of the aqueous medium from said solid residue.

3. The process of claim 1, wherein the beryllium feed source includes beryl.

4. The process of claim 3, wherein said beryl is directly introduced to said reaction stage.

5. The process of claim 3, wherein said reacting is performed at a temperature below 250°C.
6. The process of claim 3, wherein said reacting is performed at a temperature below 200°C.
7. The process of claim 3, wherein said reacting is performed at a temperature below 180°C.
8. The process of claim 3, wherein said reacting is performed at a temperature below 150°C.
9. The process of claim 3, further comprising the step of:
 - (d) introducing a second beryllium source, prior to step (c), so as to dissolve additional beryllium values and to consume at least a portion of any excess acid from step (b).
10. The process of claim 9, wherein said second beryllium source includes a readily soluble beryllium feed source.
11. The process of claim 9, wherein said second beryllium source is a readily soluble beryllium feed source.

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FIGURE 1

